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A STATISTICAL EVALUATION OF THE PYROTECHNICS ELECTROSTATIC SENSITIVITY TESTER

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OBJECT

(a) To establish optimum operating conditions for the electrostatic sensitivity tester by determining statistically which design factors contribute most significantly to its performance.

(b) To determine whether electric spark sensitivity results obtained through use of this instrument on samples of fine (29 micron) magnesium powder are reproducible.

SUMMARY

An electrostatic sensitivity tester developed at Picatinny Arsenal was evaluated statistically. The factors found to contribute most significantly to optimum instrument operating conditions were resistance, humidity, energy, and the relationship of energy to resistance. The electrostatic sensitivity results obtained with fine magnesium powder specimens were found to be reproducible. It was concluded that further work should be conducted on a variety of samples to determine the effect of various characteristics of the circuit and the maximum energy input which will produce no burning in a specified number of trials. A method for measuring this can be developed by studying the lower tails of the spark sensitivity curves. Deviations in the lower tails of the curves, which are unique for each material, are the best indicators of the materials' sensitivity characteristics.

INTRODUCTION

Previously constructed electrostatic sensitivity testers were found to have one major shortcoming. The energy delivered to the sample was inconsistent because of losses within the system, and reproducibility of results was therefore erratic. An investigation of electrostatic sensitivity testers in use by the Bureau of Mines, the Naval Ordnance Laboratory, and the British armed forces was undertaken (Refs 1, 2, 3, and 4), and a modified apparatus was constructed in an attempt to eliminate this deficiency.

The action of the pyrotechnics electrostatic sensitivity tester developed at Picatinny (Fig 1, p 12) is extremely simple. A sample is placed in the sample holder and a movable probe having a sharp point is raised above it. The apparatus is then set at the desired voltage and R–C resistance. A chosen capacitor (charged to the desired voltage) is connected between the probe and the sample holder base. The capacitor is discharged by allowing the probe to fall to a fixed distance above the sample. The operator then observes and records the resulting action.

This is a classical experiment, as many such devices have been used in the past. However, despite its apparent simplicity, it has not, in past work, consistently produced satisfactory results. Because it has a built-in resistance, capacitance, and probe-down-time mechanism (Fig 2,

p 13), the new device offers better opportunity for consistent results. One unfortunate difficulty, however, is that the probe (Fig 3, p 14) tends to become loosened by vibration, causing the operator to lose time in resetting it. After preliminary tests have been conducted, improvements to eliminate this fault will be made.

Because a large volume of data has been collected in determining optimum instrument operating conditions, it was considered desirable to issue a report on this phase of the investigation. Electric spark sensitivity data on various pyrotechnic, propellant, and explosive materials will be included in subsequent reports.

Difficulties inherent in the study of this instrument are:

1. Only attribute (Go, No go) type data can be obtained. This type of data yields only a small amount of information per observation.

2. The property of the materials to be tested is sensitivity to electric spark. This property requires a test of increased severity which is a type of test that yields little information per observation.

3. The effects of a large number of variables are determined simultaneously.

4. The spark sensitivity of a large number of materials must be evaluated. The input energy and the effect of instrument variables for any given material are of little value in the study of spark sensitivity of other materials.

5. Because of the nature of the data, non-parametric methods of analysis must be used. These methods are less efficient than parametric methods of analysis.

To reduce these difficulties to a minimum and extract the maximum amount of information possible, statistically designed experiments called factorial experiments were used. This type of designed experiment is the most efficient known. It is possible in factorial experiments to study more than one variable at a time. In general, the efficiency of the experiment is increased when a greater number of variables are studied simultaneously (Refs 5 and 8).

EXPERIMENTAL DESIGN AND ANALYSIS

Since the equipment used in this experiment was new, little was known at the outset concerning either the magnitude of the input energy required to cause burning or the effects of such other variables as might be present in the system. Therefore, a sequential approach to the problem was adopted. In this manner, something was learned about the magnitude of the input required, and it was possible to examine the results of small experiments before doing further work. The results of these exploratory experiments were not included in this report

because their contribution was mainly to eliminate "rough spots" in the apparatus.

The data was analyzed by the Kruskal-Wallis rank-sum test, sometimes called the H-Test. In determining the significance of the main effects, this test was used in the usual way (Ref 5), to determine differences among means. In determining the significance of the first-order interactions, the appropriate main effects were subtracted from each total interaction effect.

In these exploratory studies, fine (29 micron average particle size) magnesium powder[1] was used, since it was a convenient homogeneous material.

Experiment 1 (Energy Changes)

To obtain a first estimate of the input energy required, tests of increased severity were conducted using the run-down method (Refs 6 and 7). In these tests, all variables were held constant at convenient levels, except energy (in joules), which was varied by varying the capacitance. When the results were plotted on probability paper (Figs 4 through 9, pp 15 through 20), they yielded essentially straight lines, which indicated that the data could be considered, for all practical purposes, to be normally distributed. This was an important finding since it simplified interpretation of the results. The average values from these graphs (the 50% points in terms of energy) were helpful in establishing the input energy level used as a standard in subsequent experiments.

Experiment 2 (Gap Length, Humidity, Voltage, and Resistance)

The results of Experiment 1 were as follows:

1. The effects of sample size were insignificant.

2. Only inconclusive data was obtained on the effects of gap length and humidity.

3. The data obtained indicated that more should be known about the effects of voltage and resistance.

On the basis of the above findings, Experiment 2 was designed as a 4-factor complete factorial experiment to determine the effects of humidity, gap length, voltage, and resistance. The energy level was adjusted to 0.100 joule, to provide a usable distribution of successes and failures. The experiment was repeated 5 times (Tables 1, 2, and 3, pp 7, 8, and 9).

Experiment 3 (Energy, Capacitance, and Voltage)

It was clear from the 4-factor experiment that the greatest number of ignitions were being obtained by eliminating the resistance (which is connected in series between the capacitor and the probe). It now appeared desirable to determine the

[1] Sample 142, barrel No. 30, Golwynne Chemical Company

effect of voltage at different energy levels. For this purpose, a 3-factor factorial experiment was designed (Tables 4 and 5, pp 10 and 11) involving 3 levels of voltage, 6 levels of energy, and 2 levels of resistance. Resistance was included to confirm the conclusions reached in the 4-factor experiment regarding the effect of resistance.

RESULTS

Experiment No. 1

The tests of increased severity showed averages (50% ignitions) and standard deviations (slopes), in joules, as follows:

	Average	Std Dev
Figure 5	0.100	0.075
Figure 7	0.134	0.055
Figure 9	0.144	0.064

Experiment No. 2

The results of the 4-factorial statistical analysis detailed in Tables 1, 2, and 3, pp 7, 8, and 9, were:

Main Effects[a]	Effect
Voltage (V)	Not Significant
Resistance (R)	Significant[b]
Gap Length (G)	Not Significant
Humidity (H)	Significant[b]

[a]Taken from the Analysis of Variance in Table 3 (p 9)

[b]Significant at the 95% confidence level

Interactions[a]	Effect
V × G	Not Significant
R × G	Not Significant
V × H	Not Significant
R × H	Not Significant
G × H	Not Significant
V × R	Significant[c]

[c]Very highly significant, beyond the 99.9% level

Experiment No. 3

Figure 10 (p 21) represents percentage of hits (burnings) versus volts versus joules and Figure 11 (p 22) shows percentage of hits versus joules for 3000, 4000, and 5000 volts. The curve in Figure 12 (p 23) is a composite of the 3 curves in Figure 11. Tables 4 and 5 show that, while resistance (R) and energy (E) are both very highly significant, voltage (V) is not significant. Figure 12 shows the average to be 0.062 joule and the standard deviation to be 0.019 joule over the three voltage levels used.

DISCUSSION OF RESULTS

Elimination of the danger of accidental electrostatic initiation is a major reason for measuring the electric spark sensitivity of pyrotechnics, explosives, propellants, and other materials. For this purpose, instrument operating conditions that will produce the maximum burning

rate at all energy levels can be considered optimum.

From Tables 1 and 4 (pp 7 and 10), it is clear that removing all resistance from the system produces a significantly greater burning rate at all energy levels. Zero resistance can therefore be considered the optimum resistance condition for magnesium powder.

The data in Tables 4 and 5 and Figure 11 (pp 10 and 11 and 22) shows that, for zero resistance, the effect of changing the voltage from 3000 to 5000 volts is not significant. The effective sample size for evaluating the effect of voltage is 30 trials at each voltage level. Hence, the conclusion that the effect of voltage at zero resistance is insignificant at all energy levels is based on a sample size sufficient to give very good precision.

The data (Tables 4 and 5 and Figure 12 (pp 10 and 11 and 23) also makes evident a correlation between increasing percentages of burnings and increasing energy (joules).

Information on gap length and humidity is given in Table 1 (p 7). This table shows that, over the 5 resistance levels, the effect of changing the gap length from 0.01 to 0.02 inch is nil and the effect of changing the humidity from 30% to 80% is significant. The results shown in this table are considered to be reliable because they meet the effective sample size requirement for gap length and humidity, which is 250 trials at each level.

Additional work should be done to define the electric spark sensitivity of pyrotechnics, explosives, propellants, and other materials in terms of the characteristics of the electric circuit used and the maximum energy input which produces no burning in a specified number of trials. Once this definition has been developed through experience with representative materials, a method for measuring this property can be developed. This can be done by studying the lower tail of each sensitivity curve shown as a broken line in Figure 12. Since errors in this portion of the curve are rather large, it is dangerous to extrapolate from present data. In addition, significant deviations from normality can be expected. These deviations cannot be predicted by any known means. However, past experience with the impact sensitivity of explosives has shown that these deviations in the lower tail of the sensitivity curve are unique for each material and are the best indicators of sensitivity characteristics.

Work should also be carried out to determine optimum instrument conditions for pyrotechnics, explosives, propellants, and other materials. It may be possible to classify most materials into a few general types for this purpose, so that only a few instrument settings will be required. If this is not possible, then a rapid method should be developed for determining optimum conditions for new materials.

CONCLUSIONS

1. The maximum burning rate of magnesium powder cannot be obtained over the

range of energy levels surveyed if resistance is added in series between the capacitor and the probe. Varying the voltage between 3000 and 5000 volts has no effect on the number of ignitions of magnesium powder at any energy level when the resistance level is held constant.

2. Ignition is dependent on the energy released by the electrostatic sensitivity apparatus. For magnesium powder, the percentage of burnings increases with increasing energy (joules).

3. There is highly significant interaction between resistance and voltage, that is, the effect of voltage is dependent upon the level of resistance employed. Thus, any statement concerning the effect of voltage on burnings must specify the level of resistance.

4. The electrostatic sensitivity results obtained for 29-micron-average-particle-size magnesium powder are reproducible.

5. Additional work will be needed to evaluate the effect of gap length and humidity at zero resistance and to determine the electric spark sensitivity of a wide range of pyrotechnics, explosives, and propellants.

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TABLE 1

Four-Factor Factorial Electrostatic Sensitivity Experiment[a] (Experiment 2) for 29-Micron Magnesium Powder

Relative Humidity	Gap Length, inches	Resistance, kilo ohms[b]	E_1 .0222 mfd[c] 3000 volts[d]	E_2 .0163 mfd 3500 volts	E_3 .0125 mfd 4000 volts	E_4 .0099 mfd 4500 volts	E_5 .0080 mfd 5000 volts	Total Hits
25–40%	0.021	0	11111	11111	11111	01111	11111	24
"	"	90	10011	10110	11000	01011	10110	14
"	"	170	11001	01011	11100	10010	10101	14
"	"	260	01011	10101	11100	10110	01110	15
"	"	350	11011	00111	10011	10101	01100	15
"	.010	0	11111	11111	11111	11111	11111	25
"	"	90	11101	11001	00011	00101	10010	13
"	"	170	01100	00101	10010	01110	01110	12
"	"	260	11010	01110	10101	01111	11101	17
"	"	350	10010	01001	00101	01111	00111	13
75–95	.021	0	11111	11111	11111	11111	11111	25
"	"	90	11110	10111	00110	10110	11001	16
"	"	170	11011	01111	10110	11110	01011	18
"	"	260	11110	01101	10011	11010	10111	17
"	"	350	01111	11010	10110	11101	11010	17
"	.010	0	11111	11111	11111	11111	11111	25
"	"	90	10111	11101	10011	01010	11110	17
"	"	170	11101	11111	10001	10101	11010	17
"	"	260	10110	10101	11010	10001	00111	14
"	"	350	11011	11100	11101	11111	11100	19

[a]Energy, $E = \frac{1}{2} C V^2 = 0.100$ joule at every level; probe dwell-time 2.5 seconds; 2 standard scoop quantities. 0 = No Reaction; 1 = Reaction.

[b]From R–C resistance (See Table 2, p 8).

[c]Capacity

[d]Voltage

TABLE 2

Summary of Table 1 Data

	Trials	Hits	Misses
Capacitance and Voltage*			
E_1	100	75	25
E_2	100	71	29
E_3	100	63	37
E_4	100	69	31
E_5	100	69	31
Resistance, ohms			
0	100	99	1
90,000	100	60	40
170,000	100	61	39
200,000	100	63	37
350,000	100	64	36
Gap Length, inches			
.021	250	175	75
.010	250	172	78
Humidity, %			
25 to 40	250	162	88
75 to 95	250	185	65

	E_1	E_2	E_3	E_4	E_5
*Capacitance, mfd	.0222	.0163	.0125	.0099	.0080
Voltage	3000	3500	4000	4500	5000

Energy was in all cases .100 joule.

TABLE 3

Non-Parametric Analysis of Variance of Table 1 Data

	Calculated H-value[a]	Degrees of Freedom	Critical Chi-Square
MAIN EFFECTS			
Voltage (V)	3.3	4	9.49
Resistance (R)	11.7[b]	4	9.49
Gap Length (G)	0.0	1	3.84
Humidity (H)	4.8[b]	1	3.84
INTERACTIONS			
V × G	2.3	9	16.92
R × G	12.5	9	16.92
V × H	0.0	9	16.92
R × H	14.5	9	16.92
G × H	2.0	3	7.81
V × R	85.9[c]	24	36.42

[a] $H = \frac{12}{N(N+1)} \sum_{1}^{k} \frac{(R_i)^2}{n_i} - 3(N+1)$. This H-test is the Kruskal-Wallis rank-sum non-parametric test for the difference among means of counted data where H has a Chi-square distribution and

N = Total number of determinations in all groups ($\Sigma n_i = N$)

k = Number of groups

n_i = Number of determinations in an individual group

R_i = Sum of the ranks in an individual group.

[b] Significant at the 98% level

[c] Very highly significant

TABLE 4

Three-Factor Factorial Electrostatic Sensitivity Experiment[a,b] for 29-Micron Magnesium Powder

Energy, joules	Capacitance, microfarads	Voltage, kilovolts	Trials[c]	Total Hits
0.10	0.0222	3	1 1 1 1 1 1 1 1 1 1	10
"	.0125	4	1 1 1 1 1 1 1 1 1 1	10
"	.0080	5	1 1 1 1 1 1 1 0 1 1	9
.08	.0178	3	1 1 1 1 1 1 1 0 1 1	9
"	.0100	4	1 0 1 1 1 0 1 1 1 1	8
"	.0064	5	0 1 0 1 1 1 1 1 1 1	8
.07	.0155	3	1 1 1 1 0 1 1 0 1 1	8
"	.0088	4	0 1 1 1 1 0 0 1 1 1	7
"	.0056	5	1 1 1 1 0 1 0 1 0 1	7
.06	.0133	3	1 1 0 1 0 1 0 0 1 0	5
"	.0075	4	0 1 0 0 0 1 0 1 1 1	5
"	.0048	5	0 1 0 1 0 0 1 0 1 0	4
.05	.0111	3	0 0 1 0 0 1 0 1 1 0	4
"	.0063	4	0 0 1 0 0 1 0 0 0 1	3
"	.0040	5	0 0 0 0 0 0 1 0 0 0	1
.04	.0089	3	0 0 0 1 0 1 0 0 0 1	3
"	.0050	4	0 0 0 0 0 0 0 0 0 0	0
"	.0032	5	0 0 0 0 0 0 0 0 0 0	0

[a]This experiment was repeated for 10,000 ohms R–C resistance with 100% failures (No reactions). See Table 5 (p 11).

[b]Probe dwell-time 2.5 secs, R–C resistance = 0 ohms, Gap length 0.01 to .02 in.; R. H. 25 – 35%

[c]0 = No reaction; 1 = Reaction

TABLE 5

Summary of Table 4 Data (See also Figs 8 and 9)

Energy, joules	Voltage	% Hits Zero Resistance	% Hits 10,000 ohms Resistance
.10	3000	100	20
.10	4000	100	0
.10	5000	90	0
.08	3000	90	0
.08	4000	80	0
.08	5000	80	0
.07	3000	80	0
.07	4000	70	0
.07	5000	70	0
.06	3000	50	0
.06	4000	50	0
.06	5000	40	0
.05	3000	40	0
.05	4000	30	0
.05	5000	10	0
.04	3000	30	0
.04	4000	0	0
.04	5000	0	0

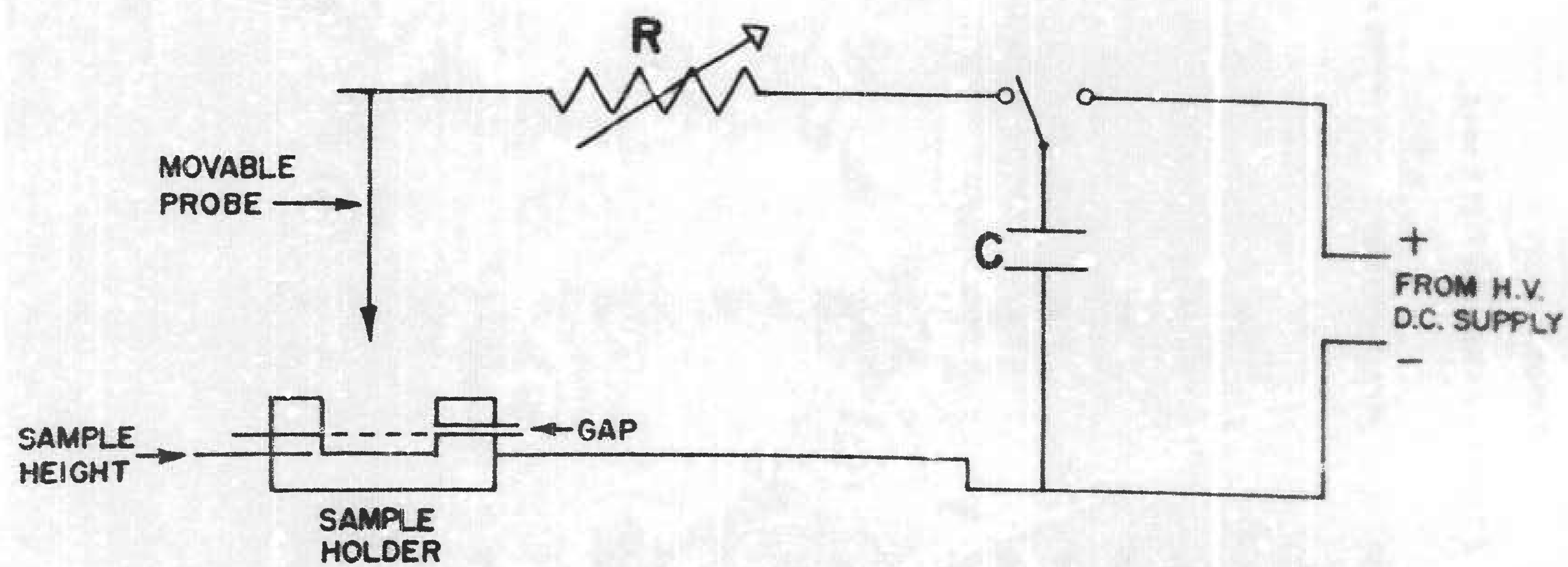

Fig 1 Schematic of Electrostatic Sensitivity Test Apparatus

Fig 2   Pyrotechnic Electrostatic Sensitivity Tester

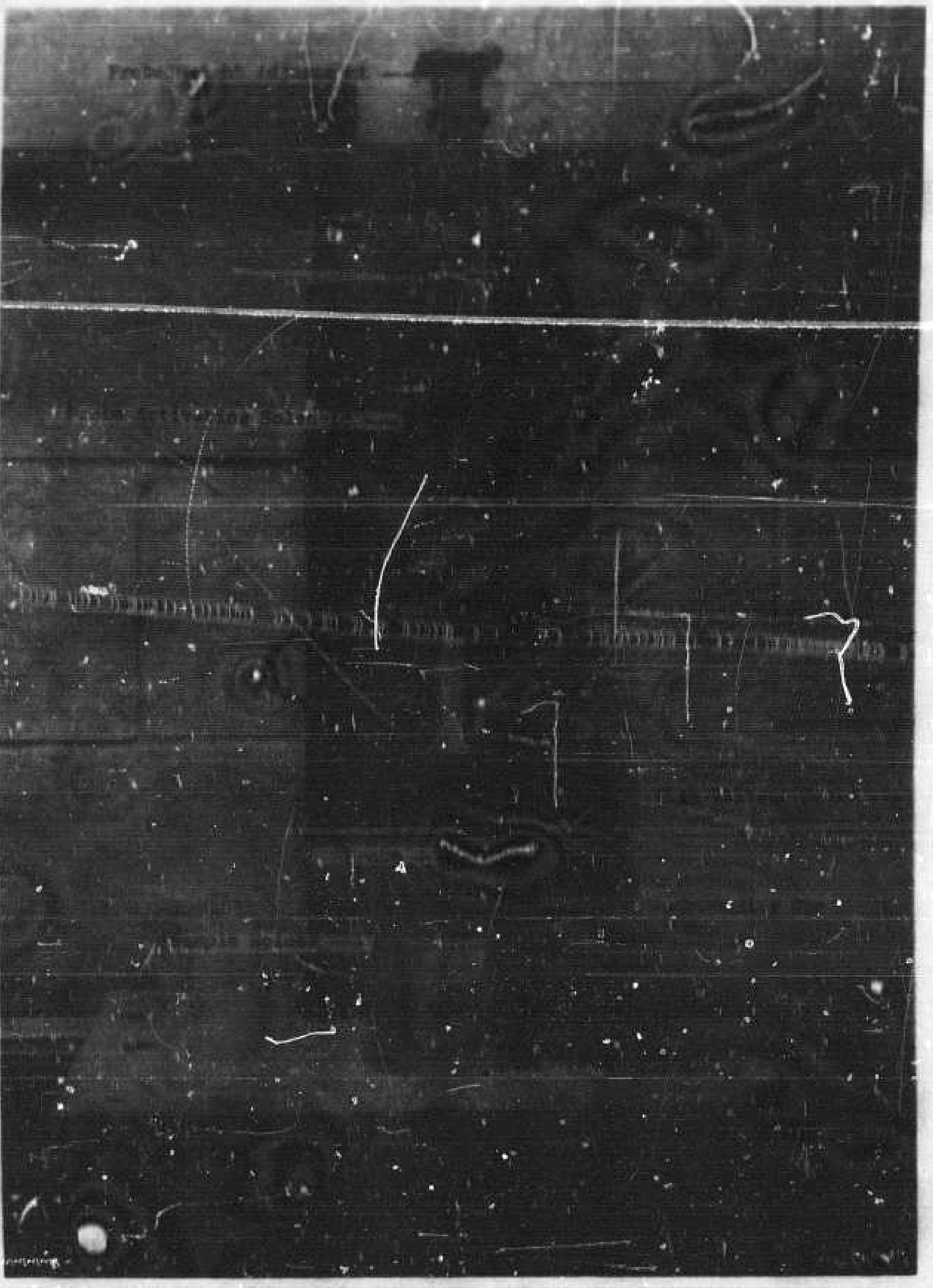

Fig 3 Sparking Mechanism, Probe, and Adjustments

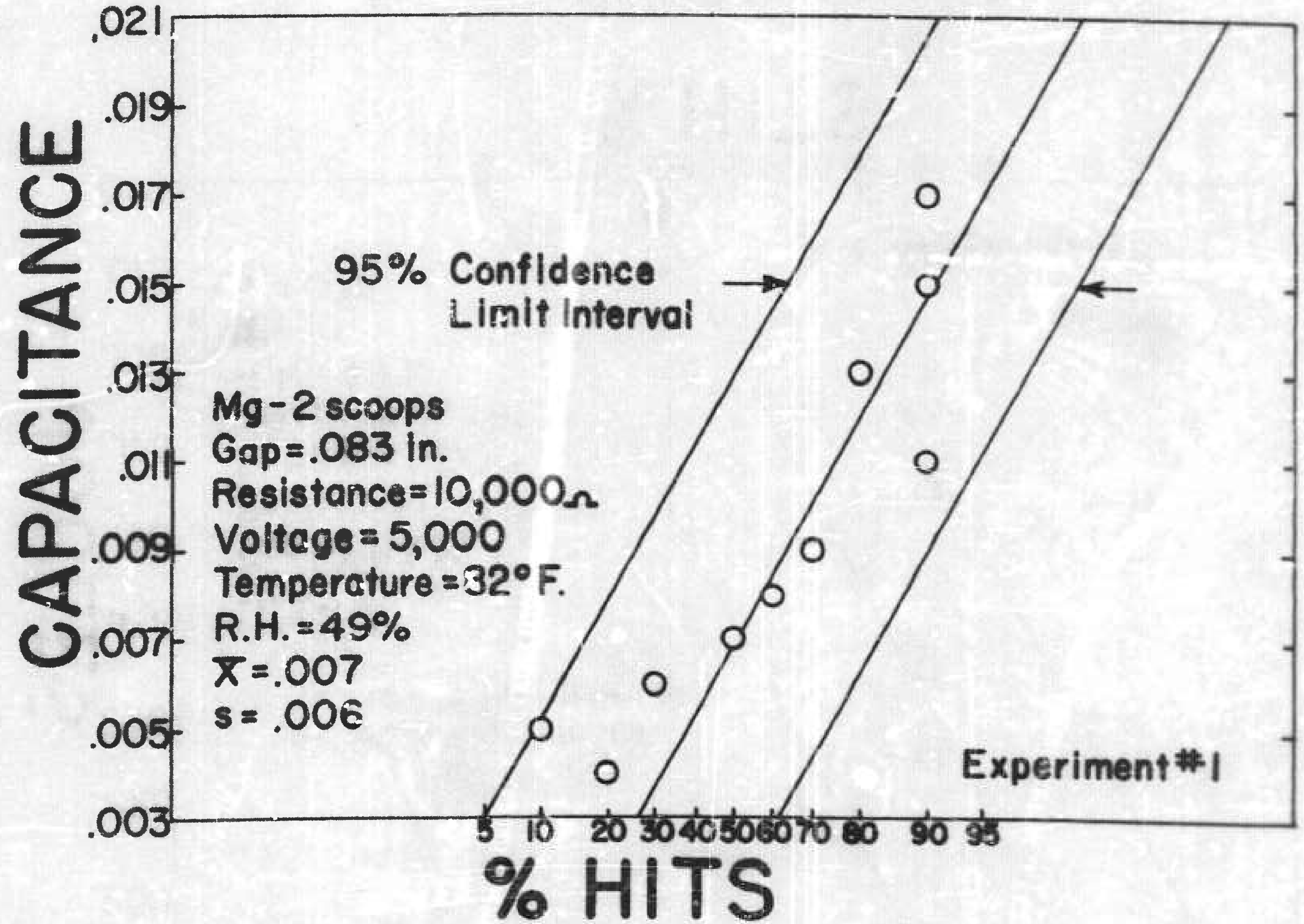

Fig 4 Normality of Distribution of Experiment 1 Capacitance Data

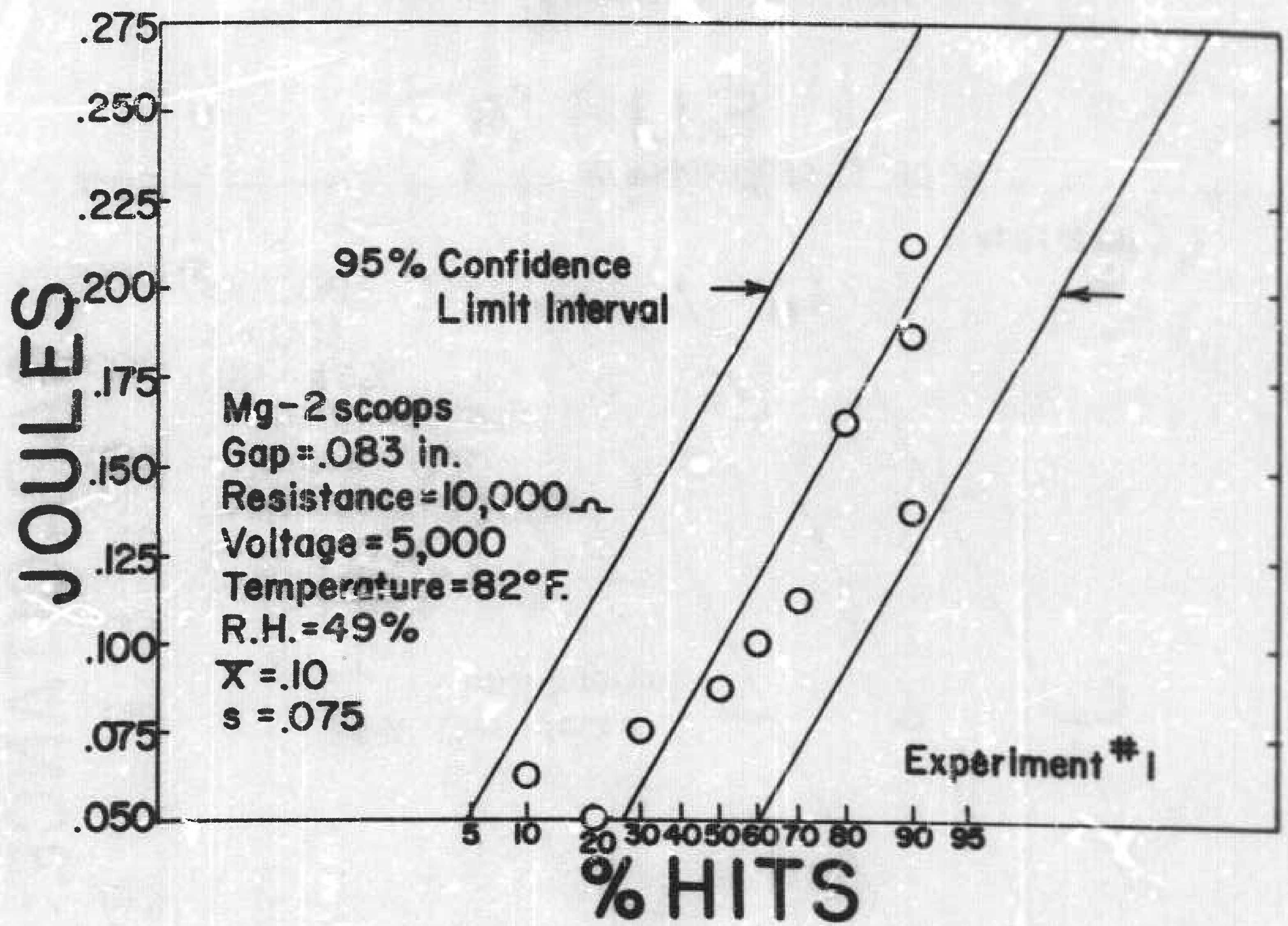

Fig 5 Normality of Distribution of Experiment 1 Energy Data

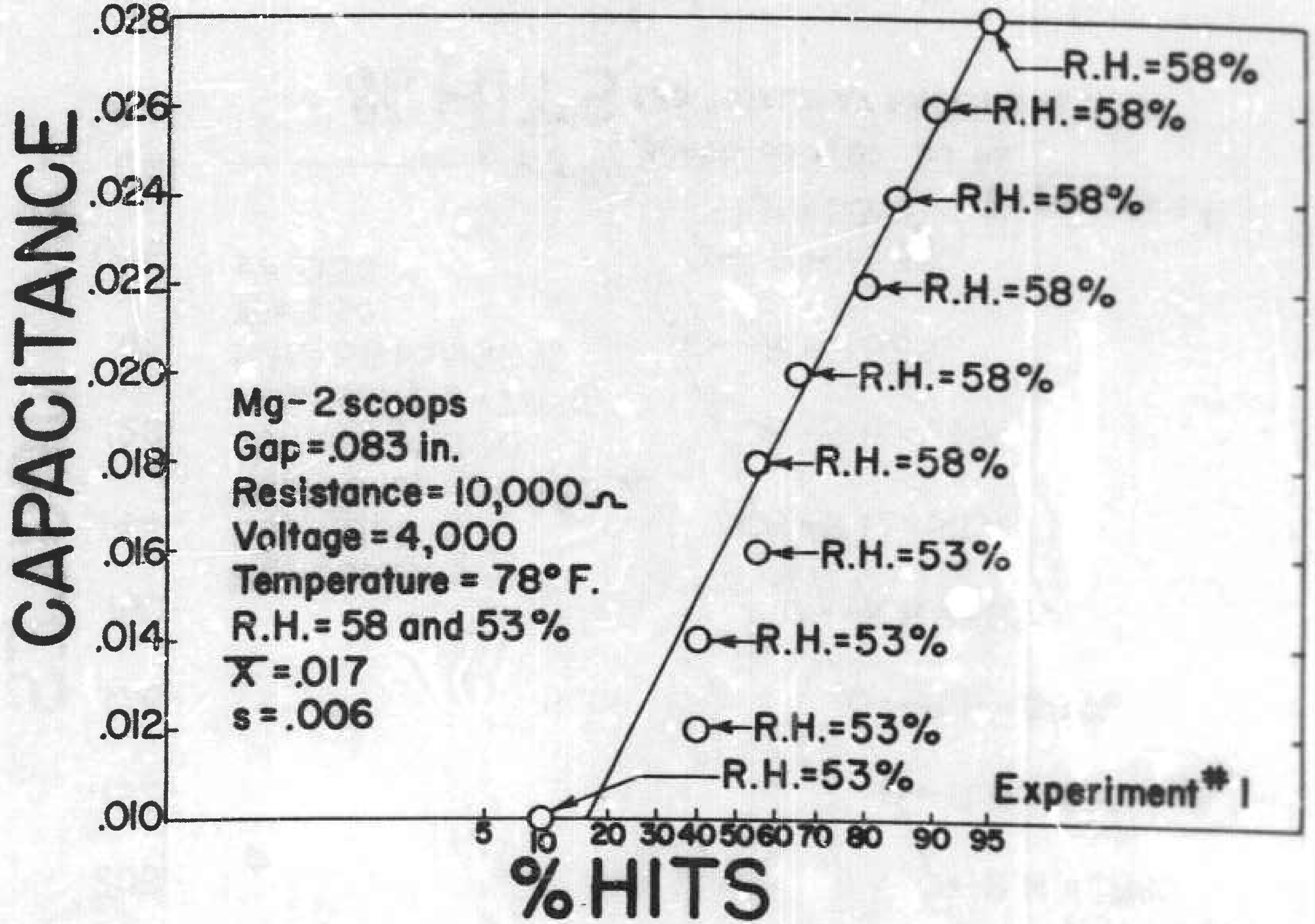

Fig 6 Preliminary Data on Effect of Humidity on Capacitance

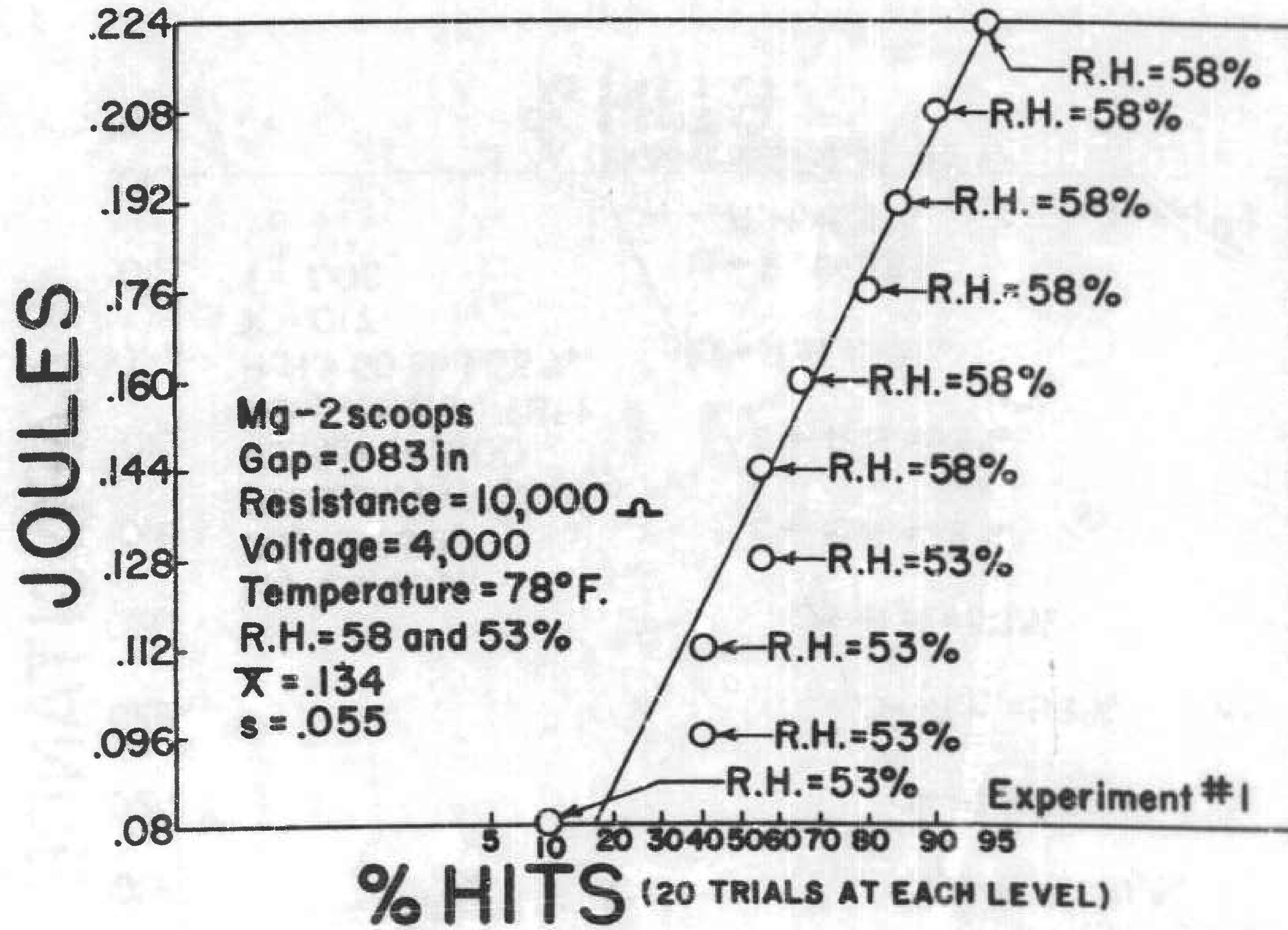

Fig 7 Preliminary Data on Effect of Humidity on Energy

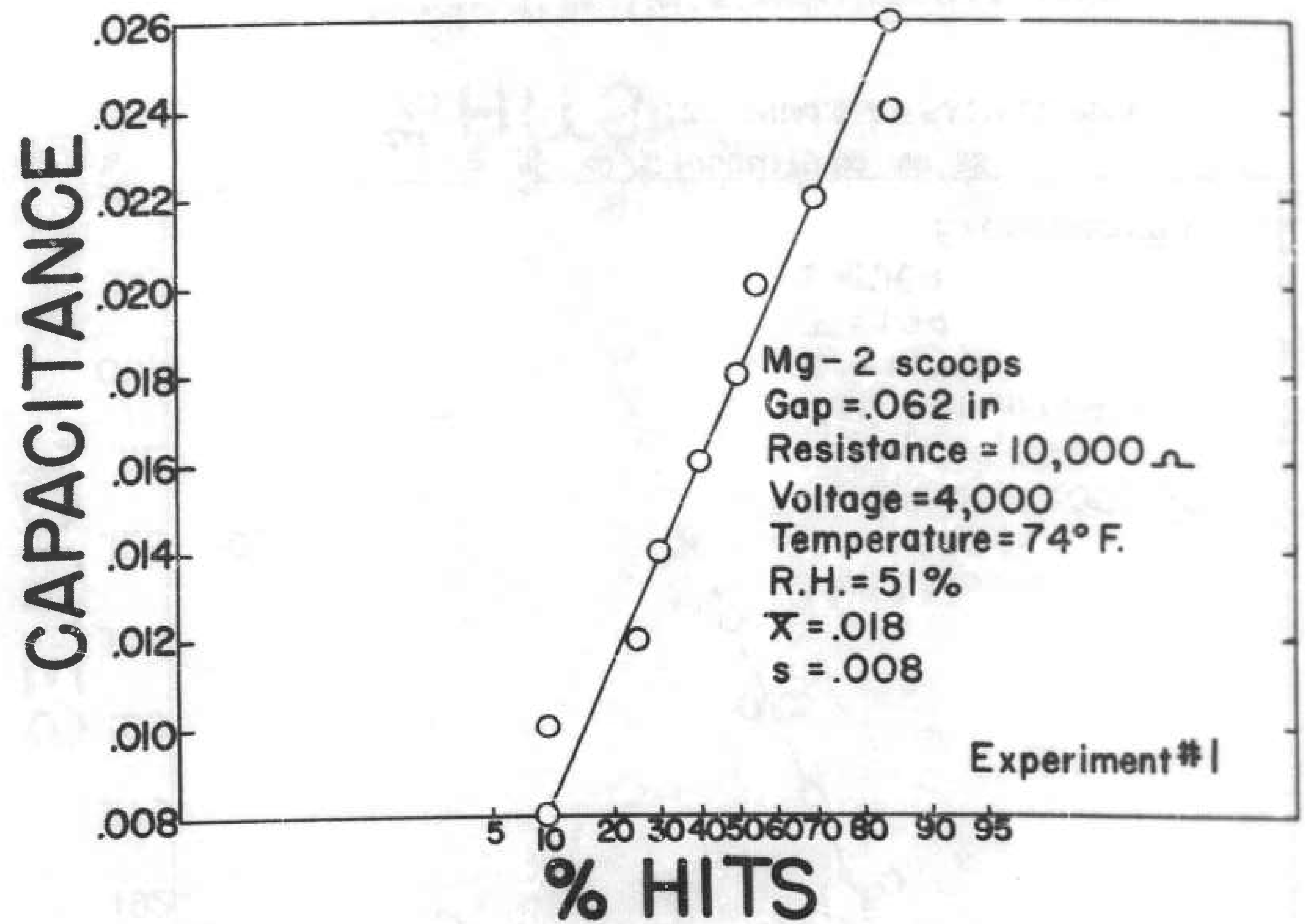

Fig 8 Confirmation of Figures 4 and 6 at Controlled Temperature and Humidity

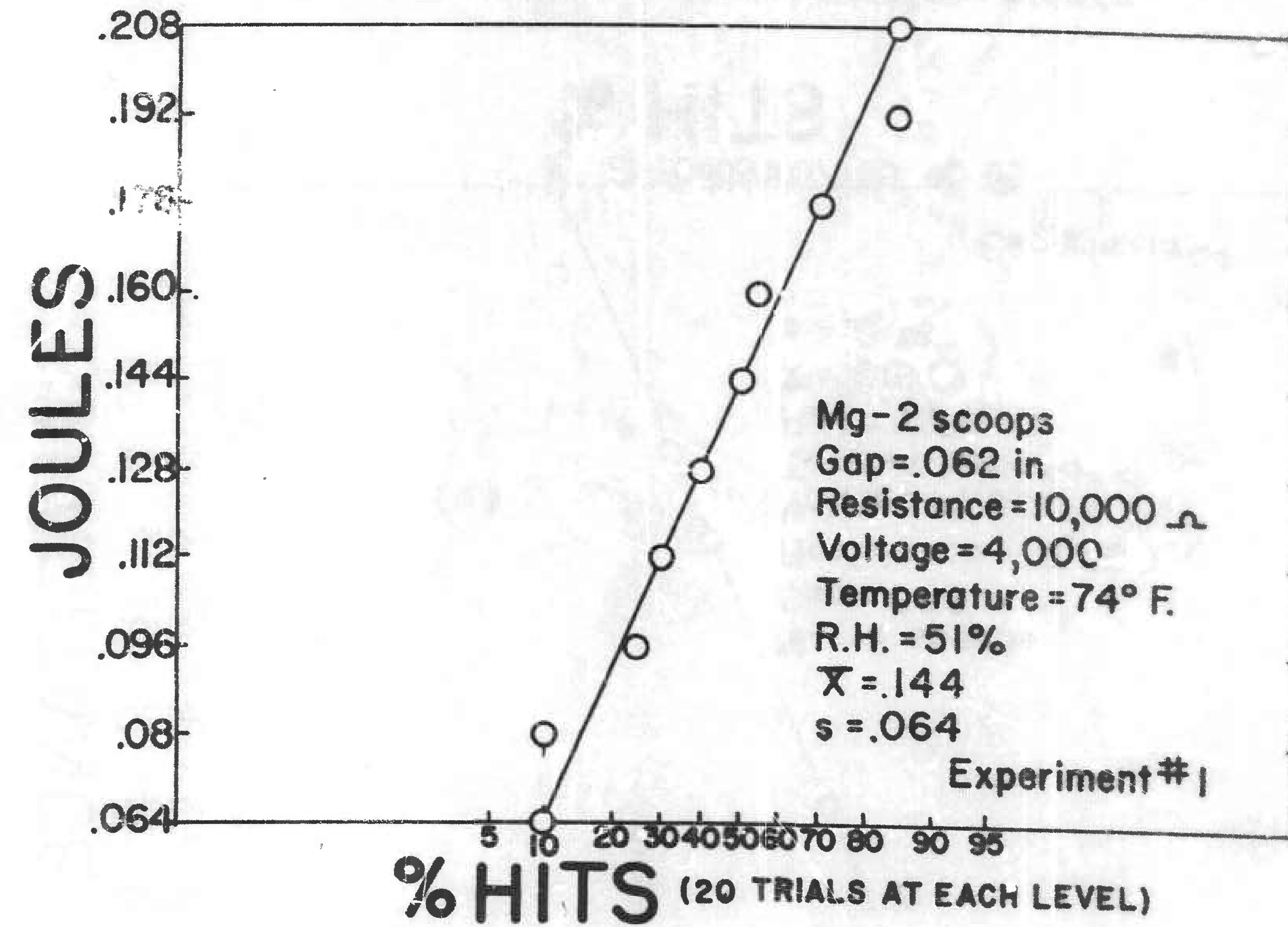

Fig 9 Confirmation of Figures 5 and 7 at Controlled Temperature and Humidity

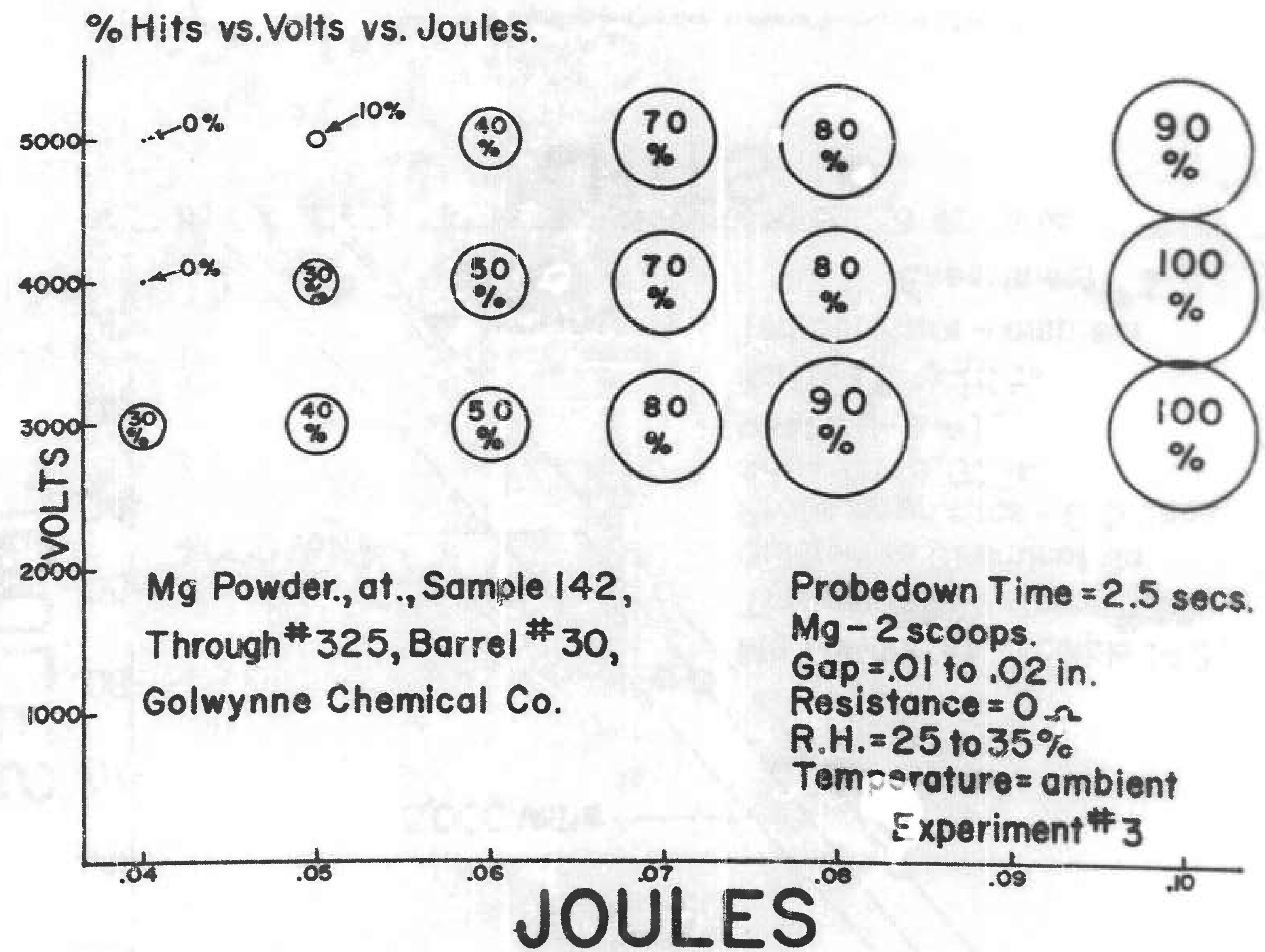

Fig 10 Area Graph Showing Interaction Between Voltage and Energy

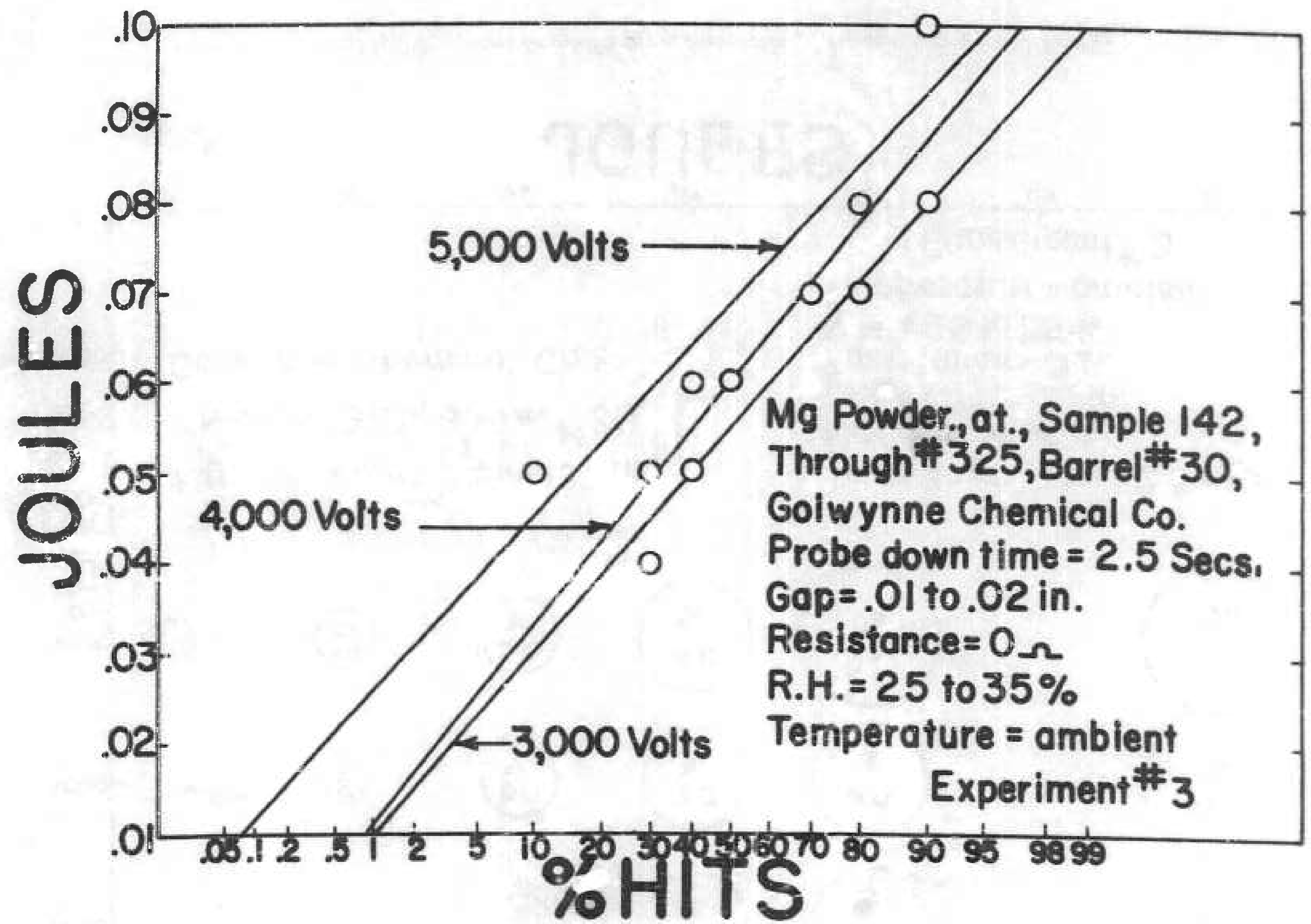

Fig 11 Line Graph Showing Interaction Between Voltage and Energy

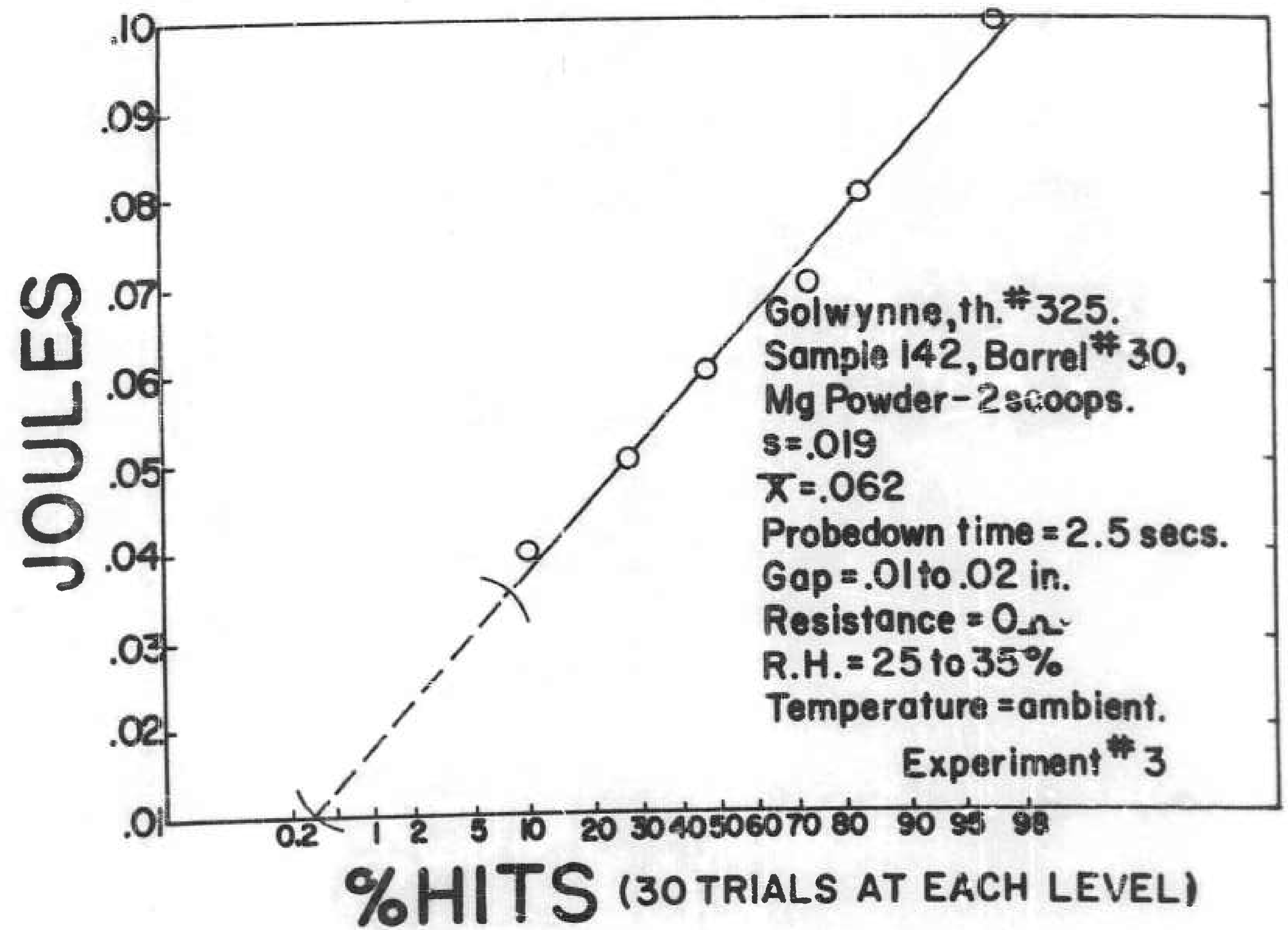

Fig 12 Composite of Figure 11 Curves